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Task Technical and Quality Assurance Plan for 804 UG Organic Stripping

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LIST OF ACRONYMS

AD	Analytical Development
DDI	Distilled deionized water
DOE	Department of Energy
DWPF	Defense Waste Processing Facility
HLW	High Level Waste
IC	Ion Chromatography
MST	Monosodium Titanate
SEM	Scanning Electron Microscope
SRS	Savannah River Site
SRNL	Savannah River National Laboratory
SPF	Saltstone Production Facility
SVOA	Semi-Volatile Organic Analysis
VOA	Volatile Organic Analysis

1.0 Introduction

The SRS Decontamination and Decommissioning (D&D) Organization is evaluating options to disposition the 804 underground tank which includes removal of the sludge from this tank and requested assistance from SRNL personnel to develop methods to effectively mobilize the sludge. However, analysis of the tank constituents has indicated a large quantity of flammable organics is present in the tank, intermixed with a large quantity (>1 fissile mass of Pu^{239}) of actinide-containing sludge.[1] The presence of the organics presents a problem for disposition of the tank to the HLW tanks. A brainstorming session on 11-22-05 discussed several possible strategies to remove the organics from the tank. SRNL was tasked with performing tests to study the various organic removal options on Tank 804 sludge.[2]

The objectives of this task follow.

- Experimentally simulate evaporation and steam stripping of the tank volatile components, and sample the distillate over time for the presence of organic species. CURRENTLY ON HOLD
- Evaluate filtration as a means of removing organic species after poisoning with Mn and the neutralization process (does organic remain with the solids?)

In addition, Site D&D has requested an evaluation of Pu distribution in the caustic adjusted 804 sludge without manganese addition at pH• 14 (1.0-1.2M NaOH).[3] CURRENTLY ON HOLD

2.0 Task Description

2.1 Deliverables and Acceptance

The deliverables include a technical report. The report will include a design check per WSRC Manual E7, procedure 2.60. The final reports will receive approval from selected personnel.

2.2 Requirements

All work will comply with established quality assurance procedures identified in this document. Approval of the document indicates acceptance of the plan.

2.3 Prerequisites

- CS&T will complete the SRNL Conduct of Research and Development hazards assessment checklist prior to starting work.
- CS&T will prepare and issue a Task Technical and Quality Assurance Plan that is approved by the appropriate SRNL Management, SRS D&D Management, and SRNL-QAD.

- CS&T will ensure the M&TE are calibrated prior to starting the work.
- CS&T will arrange waste disposal.

3.0 Activities

3.1 Approach

SRS D&D provided SRNL with a sample of the sludge from Tank 804. This material will be used in the evaporation, filtration, and pH adjustment tests. Even though there is a possibility that hazardous organo-mercurials will be formed, we are not explicitly trying to qualify or quantify them.

3.1.1 Evaporation/Steam Stripping Tests (CURRENTLY ON HOLD)

A 1-L batch of sludge will be prepared in the same manner that the sludge would be prepared for the tank farm.

- A mass of 804 sludge will be mass diluted 10:1 with DDI water
- Enough $Mn(NO_3)_2$ will be added to raise the solution to 1M in manganese (40:1 Mn:^{239}Pu by wt)
- The mixture will be pH adjusted to a free hydroxide concentration of 1.2 M (pH 14)

The resulting adjusted slurry will be bottled, analyzed for Mn, Pu, VOA and SVOA in duplicate and set aside for use in the tests. Mixing the slurry will be done by shaking, the same method as used in WSRC-TR-2005-00540.

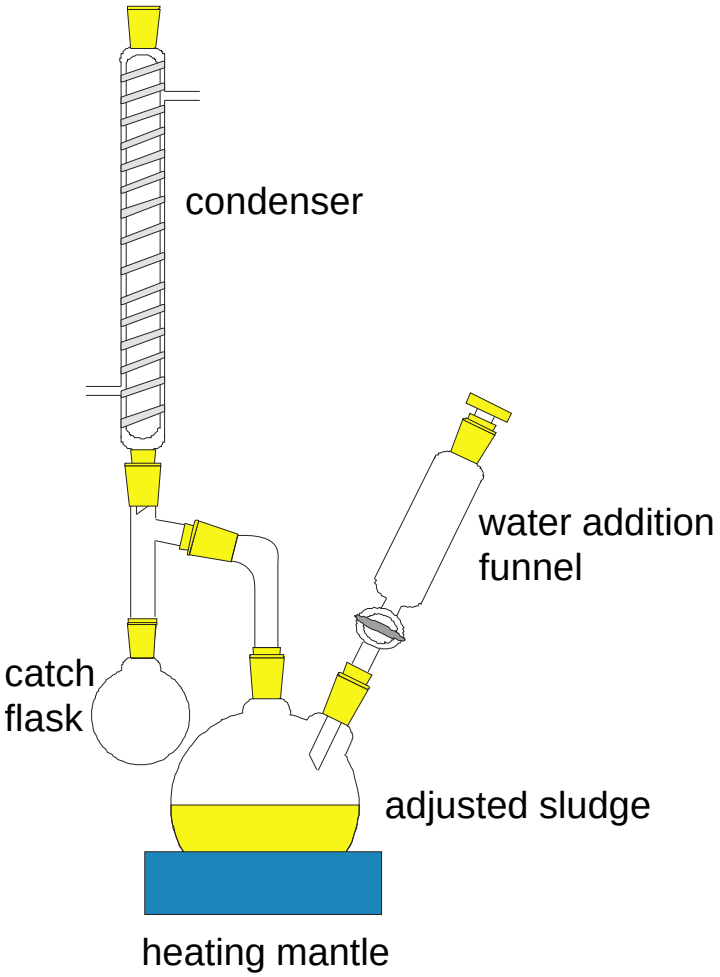
Researchers will assemble a distillation apparatus (Figure 1) for each of the tests.

The distillation flask will be a 500 mL working volume unit. For each test, 200 mL of adjusted slurry that was previously prepared will be added to the flask. This 200 mL level on the flask will be marked on the flask so the operator can determine if water should be added to the flask. We need to maintain a fixed volume in the flask to avoid distilling the sludge to dryness. As water leaves the distillation pot, water will be added back to the mark by the water addition funnel. The distillate catch flask will be marked at the 40 mL level so we know when to remove the catch flask.

SRNL envisions 2 tests in total. The first test is a short-term test run at different temperatures designed to find an adequate operating temperature. At first, the evaporation will be run at 60 ºC until a single catch flask has been filled to the 40 mL mark, or for 8 hours, whichever comes first. The amount of distillate removed will be quantified, and the overheads sent forward to be analyzed for VOA and SVOA. After replacing the catch flask with an empty one, the evaporation temperature will be raised to 80 ºC for 8 hours or until the catch flask has been filled

to the 40 mL mark. The amount of distillate removed will be quantified, and the overheads sent forward to be analyzed for VOA and SVOA. After replacing the catch flask with an empty one, the evaporation temperature will be raised to 100 ºC for 8 hours or until the catch flask has been filled to the 40 mL mark. The amount of distillate removed will be quantified, and the overheads sent forward to be analyzed for VOA and SVOA. During the test, water will be added to the distillation pot as required to maintain a constant volume. Temperature monitoring will be provided by a thermocouple in the distillation pot (not shown in Figure 1). The contents of the distillation pot will be heated using a heating mantle. Each flask will then be set aside for later analyses. As a distillate catch flask is removed, the time from the beginning of distillation will be recorded. At the end of the test, the distillation pot will be sampled and sent forward to ADS for analyses.

Figure 1. Distillation Apparatus



The second test will be run after the results of the first test are known. The most appropriate temperature (60, 80, 100 ºC) will be chosen for the final test. This test will be run for 48 hours straight (requiring around the clock coverage), with water being added to the distillation pot as required. Alternatively, the distillation could be run in six 8 hour blocks to avoid excessive personnel costs. As a distillate catch flask is filled to the mark, it will be replaced by another empty one. The ratio of overheads to starting volume to obtain adequate evaporation will be determined. At the end of the tests, the distillation pot will be sampled and sent forward along with the distillate samples to ADS for analyses (VOA, SVOA, Mn and Pu analyses). The residual hold up material in the glass joints will be flushed, collected and sent forward to ADS for VOA and SVOA analyses. Finally, a sample of the distillation pot will taken for Scanning Electron Microscope (SEM) analysis.

3.1.2 Filtration

Researchers will prepare slurry as described in the evaporation tests. The slurry will contain 1 part Tank 804 sludge and 10 parts water. To this slurry they will add sufficient manganese nitrate to produce an adjusted slurry that is 1 M in manganese. They will then add sufficient sodium hydroxide to adjust the pH to 14. After agitating (shaking) this slurry, they will filter sub-samples through various pore size filters and submit the filtrate and rejected solids for organic analysis by VOA and SVOA and Pu and Mn.

3.1.3 pH Adjustment Tests without Manganese Addition (CURRENTLY ON HOLD)

Personnel will prepare a slurry containing Tank 804 sludge diluted 10:1 with water, then adjusted with nitric acid to pH 3, and finally adjusted to pH 14 with sodium hydroxide. After agitating by shaking, they will allow the slurry to settle overnight, collect samples from the top and bottom of the container, and analyze the samples by gamma ray spectroscopy for plutonium, by peroxide fusion/plutonium triphenyltrifluoroacetone scintillation analysis (PuTTA) for plutonium, and by volatile organic analysis (VOA)/semivolatile organic analysis (SVOA) for organic species. The researchers will take pictures before and after shaking the slurry bottle.

The solution will be remixed to suspend the solids, then the researchers will filter subsamples through various pore size filters and hold the filtrate and rejected solids samples for future analysis.

3.2 Measurement and Analysis

This task will use the following M&TE:

- Balance calibration will reference weights traceable to NIST standards.

Once a distillate flask is removed from the apparatus is will be set aside and sealed for later analysis. Once ready, the flask will be sent for Volatile Organic Analysis (VOA) and Semi-Volatile Organic Analysis (SVOA). At the end of each of the two tests, the distillation pot will be sampled and sent forward for VOA, SVOA, Pu, Mn and Ion Chromatography (IC) analyses. The intention is to determine the type and quantity of organic material that has distilled over as a rough function of time as well as assure that there is no large separation of plutonium from the manganese poison. Finally, the researchers will attempt to flush the glassware for residual hold-up material in the glass joints and this material forward to ADS for VOA and SVOA analyses.

The researchers will submit the filtrate and rejected solids for organic analysis by VOA and SVOA and plutonium analysis by PuTTa and will submit samples of the pH adjusted slurry for plutonium, manganese, and organic analyses by the methods described above.

4.0 Documentation

CS&T researchers will record pertinent information in a controlled laboratory notebook in accordance with Manual L1, SRNL Procedures Manual, and Procedure 7.16. All reports will receive review and approval from SRNL line management as well as appropriate personnel within D&D. A Savannah River National Laboratory researcher, not involved in the task, will conduct a design check of the report. This design check will include reviewing the input data, the analytical and experimental approach, mathematical calculations, assumptions, and the recommendations.

5.0 Risk Review

The following table lists other risks and mitigating factors.

Risk Factor	Event	Mitigation
Equipment	Failure	Replacement or repair of item with a potential delay.
	Operation Difficulty	The distillation apparatus may be difficult to manipulate in the cells and this may impose a working delay.
Personnel	Illness, vacation	Primary and secondary researchers identified.
Facility	Outage (electrical, ventilation)	Communicate with SRNL operations so outages do not interfere with testing.

The data at 80 ºC and 100 ºC may be predicated on the changes that occur at the early stages of the experiment. Hence, the rate or amount of organic removal at 80 ºC and 100 ºC will not reflect the initial rate at those conditions but will be impacted by the earlier [lower temperature] portion of the tests.

In addition, there is a risk that loss of organics due to heating and incomplete sampling may occur and hence the values may have wider variance and may not lead to closure on the overall organic balance.

Finally, the SVOA or VOA analyses may not accurately identify of quantitate the presence of methyl mercurials or dimethyl mercury, both of which can pose work hazards.

6.0 Schedule

The following tentative schedule estimates the completion of deliverables for this task. We will accelerate this where possible.

Issue Task Plan	December 16, 2006
Complete filtration experimental and pH adjustment work	February 9, 2006
Complete evaporation experimental work	February 22, 2006
Issue report on filtration	March 13, 2006
Issue report on evaporation	March 31, 2006

The schedule for support of this task requires resource loading across all SRNL support activities. The lead investigator will provide information on schedule logic, task duration, needed resources, and resource constraints to the SRNL schedule development personnel.

7.0 Safety and Quality Assurance

The authors will complete the SRNL Conduct of Research and Development checklist prior to starting this task.

7.1 Quality Assurance

Quality Assurance Checklist

See Attachment 1.

7.2 Documents Requiring Customer Approval

The following documents require customer approval:

- Task Technical and Quality Assurance Plan
- Final Report

7.3 Records

The following items shall be designated records for this experimental program:

- The Task Technical Plan
- All applicable laboratory notebooks
- The final technical report
- Supporting documents as determined by the task leader (Tom Peters).

8.0 References

[1] L. M. Chandler, A. R. Jurgensen, D. M. Missimer, "Tanks 804, 808, & 809 Radiochemistry and Organic Data Compilation and Independent Technical Review", SRNL-ADS-2005-00463, July 29, 2005.

[2] "Determine Effectiveness of Organic Removal from Slurried Tank 804 Sludge by Steam/Heating Stripping and Filtration", R. A. Eubanks, December 6, 2005.

[3] M. R. Poirier, M. E. Stallings, P. R. Burket, and S. D. Fink, "Composition and Flow Behavior of F-Canyon Tank 804 Sludge following Manganese Addition and pH Adjustment", WSRC-TR-2005-00540, November 30, 2005.

ATTACHMENT 1. QA CHECKLIST

WPT TASK QUALITY ASSURANCE PLAN CHECKLIST

Task Technical Plan No: WSRC-RP-2005-01950, Rev. 0 Task Title: Task Technical and Quality Assurance Plan For 804 UG Organic Stripping Listed below are the sections of WSRC QA Manual (1Q). Check the 1Q sections applicable to your task. Also, check procedures CS&T implements to control the task. This checklist identifies controls for task activities performed by CS&T only. (Form Revised 11/30/2005)

WSRC 1Q Section	Applies To Task	***Procedures Implemented by CS&T***	Procedure Used
Organization	X	1Q, QAP 1-1, Organization	X
		L1, 1.02, SRTC Organization	X
	X	1Q, QAP 1-2, Stop Work	X
QA Program	X	1Q, QAP 2-1, Quality Assurance Program*	X
		1Q, QAP 2-2, Personnel Training & Qual.	X
		L1, 1.32, SRTC Read and Sign/Briefing Program	X
	X	1Q, QAP 2-3, Control of R&D Activities*	X
		L1, 7.10, Control of Technical Work	X
	X	L1, 7.16, Laboratory Notebooks and Logbooks	X
		1Q, QAP 2-4, Auditor/Lead Auditor Qual. & Cert.	NA for CS&T
		1Q, QAP 2-5, Qual. & Cert. of Independent Insp. Personnel	NA for CS&T
		1Q, QAP 2-7 QA Program Req. for Analytical Measurement Systems	
Design Control		1Q, QAP 3-1, Design Control	
		L1, 7.10, Control of Technical Work	
Procurement Document Control		1Q, QAP 4-1, Procurement Document Control	
		E7, 3.10, Determination of Quality Requirements for Procured Items	
		7B, 3E (for reference only)	
Instructions, Procedures and Drawings	X	1Q, QAP 5-1, Instructions, Procedures, & Drawings	X
		E7, 2.30, Drawings	
		L1, 1.01, SRNL Procedure Administration	X
Document Control	X	1Q, QAP 6-1, Document Control	X
		1B, MRP 3.32, Document Control	X
Control of Purchased Items and Services		1Q, QAP 7-2, Control of Purchased Items & Services	
		7B & 3E (for reference only)	
		1Q, QAP 7-3, Com. Grade Item Dedication	
		E7, 3.46, Replacement Item Evaluation/Commercial Grade Dedication	
Identification & Control of Items	X	1Q, QAP 8-1, ID and Control of Items*	X

Control of Processes		1Q, QAP 9-1, Control of Processes	NA for CS&T
		1Q, QAP 9-2, Control of Nondestructive Exam.	NA for CS&T
		1Q, QAP 9-3, Control of Welding & Other Joining Proc.	NA for CS&T
		1Q, QAP 9-4, Work Processes 1Y, 8.20, Work Control Procedure	

Inspection		1Q, QAP 10-1, Inspection L1, 8.10, Inspection	NA for CS&T
Test Control		1Q, QAP 11-1, Test Control (applies to CS&T only for acceptance testing; R&D test activities are controlled by 1Q, QAP 2-3)	
Control of Measuring & Test Equipment	X	1Q, QAP 12-1, Control of Measuring & Test Equipment	X
		1Q, QAP 12-2, Control of Installed Process Instrumentation	
		1Q, QAP 12-3, Control & Calibration of Radiation Monitoring Equipment	
Packaging, Handling, Shipping & Storage		1Q, QAP 13-1, Pkg., Handling, Ship. & Storage*	
Inspection, Test, and Operating Status		1Q, QAP 14-1, Inspection, Test, & Operating Status*	
Control of Nonconforming Items & Activities	X	1Q, QAP 15-1, Control of Nonconforming Items*	X
Corrective Action System	X X	1Q, QAP 16-3 Corrective Action Program 1.01, MP 5.35, Corrective Action Program	X
QA Records	X	1Q, QAP 17-1, QA Records Management* L1, 7.16, Laboratory Notebooks and Logbooks	X X
Audits	X	1Q, QAP 18-2, Surveillance	
		1Q, QAP 18-3, QA External Audits	
		1Q, QAP 18-4, Management Assessment Program 12Q, Assessment Manual	
		1Q, QAP 18-6, Quality Assurance Internal Audits	
		1Q, QAP 18-7, Quality Assurance Supplier Surveillance	
Quality Improvement	X	1Q, QAP 19-2, Quality Improvement*	X
Software Quality Assurance		1Q, QAP 20-1, Software QA L1, 8.20, Software Management & QA	
Environmental QA		1Q, QAP 21-1, Quality Assurance Requirements for the Collection and Eval. of Environmental Data	NA for CS&T

Exceptions/Additions-Procedures identified on the checklist with an asterisk (*) are supplemented by a SRNL clarification in L1, 8.02, “SRNL QA Program Clarifications”. WSRC-IM-2002-00011, “Technical Report Design Check Guidelines,” will be used to help ensure the quality and consistency of the technical reviewer process for technical reports produced by SRNL Waste Treatment Technology.